Particle Size Distributions: Dynamic Image Analysis Beats Laser Diffraction in Micron to Millimeter Range

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Laser diffraction is the most frequently used measurement technique for the analysis of particle size distributions in the range 1 micron to 1mm in the context of quality control. Modern laser diffraction systems offer some convincing advantages such as short measurement times, easy operation and reproducible analysis results. However, they also have various disadvantages: Even if the instruments have been calibrated and validated, an absolute particle size measurement is not possible. Various round robin tests have shown that the analysis results depend strongly on the type of instrument and even on the particular model and software version.

The laser diffraction method is further limited by the unsatisfactory detection for small outlier volume fractions (over-size and under-size) of approximately 2 to 3%, as well as the poor resolution of particles in the range from a few hundred microns to millimeters. Although laser diffraction systems are able to detect particles > 10 nanometers, only very few measuring channels are provided for particle sizes of approximately 1mm. The resolution for these particles is rather poor. Thus, it is not possible to precisely resolve multimodal size distributions, as particles of a few hundred microns size difference are classified in the same size class.

The complex, indirect measurement algorithm used by laser diffraction is like a "black box" for many users. The selection of the optimum evaluation parameters requires some experience. For the correct interpretation of the results it is often necessary to have some previous knowledge about the sample characteristics. Wrong assumptions and parameters lead to reproducible but inaccurate measurement results. Static laser light scattering is a rapid method, easy to carry out but difficult to evaluate. The ideal measurement method should directly detect the individual particle characteristics, for example by taking an image of the particle and calculating it directly.

Now such a direct measurement technology for fine powders > 1 micron is available with the new particle analyser CAMSIZER XT (see Infobox). It uses the measurement technology of the ISO 13322-2 Dynamic Image Analysis standard, and beats laser diffraction with regards to resolution and detection limits by more than a factor 10.

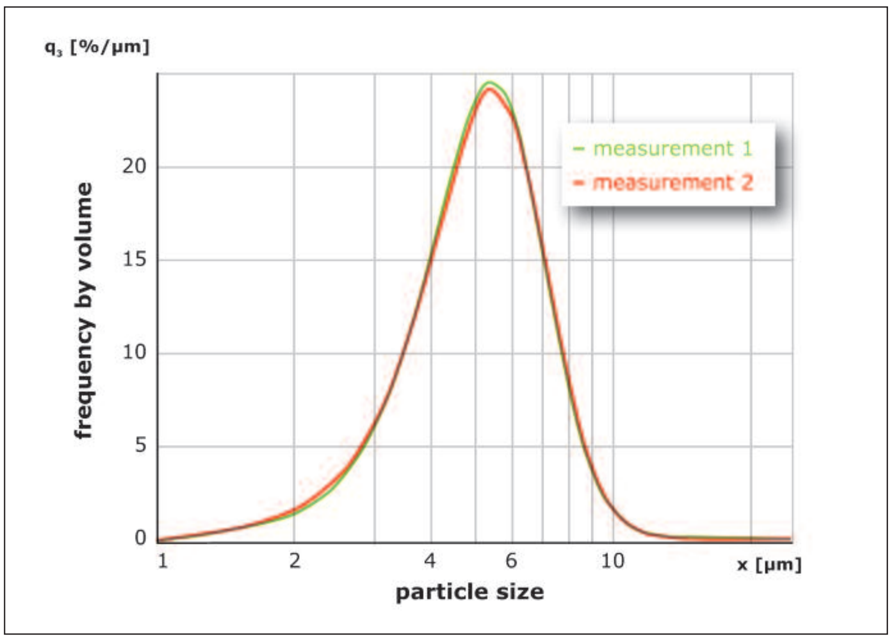

Figure 1. Measurement of silicon carbide (abrasive) with a size distribution of 1 – 10µm and a mean value of 5µm (dry dispersion with compressed air).

Until recently, dynamic image analysis was only established for the measurement of dry, pourable powders and granules in the size range above 30µm. Thanks to an advanced computer and camera technology finer particles can now be displayed more sharply and evaluated in real time. The evaluation speed achieves 275 pictures per second.

For the measuring range of 1 micron and above the image analysis method now also offers convincing benefits: as the particle images are taken directly with a camera of extremely high resolution, their size and shape can be accurately determined, even over a few orders of magnitude and consequently with a much higher resolution when compared with laser diffraction.

The following application examples show the superiority of dynamic image analysis.

Accurate Determination of Oversized Particles

The laser light scattering method always detects a particle collective, for example, the scattering signal is an average of many particles. Small amounts of undersized or oversized particles only cause a minor change in the light scattering pattern and therefore cannot be reliably detected with laser diffraction. Depending on the sample material, a volume fraction of 2 – 3% is considered as the absolute detection limit. The image analysis method, however, evaluates individual particles and detects, depending on the operation mode, every single particle of the sample. Only a few particles in the sample are enough for reliable detection, even if these particles amount to less than 0.01 % of the entire sample volume. This opens up new perspectives for the characterisation and ensures improved quality of the production monitoring process.

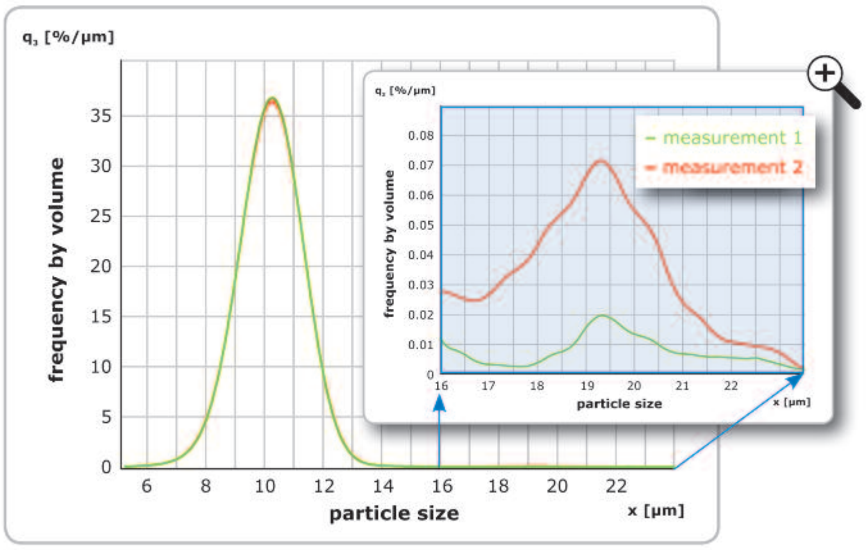

Figure 2. Comparison of two different samples with different fractions of oversized particles. Sample 2 (red) contains 0.2% more over-size at 20µm. It is impossible to detect such small differences with laser diffraction.

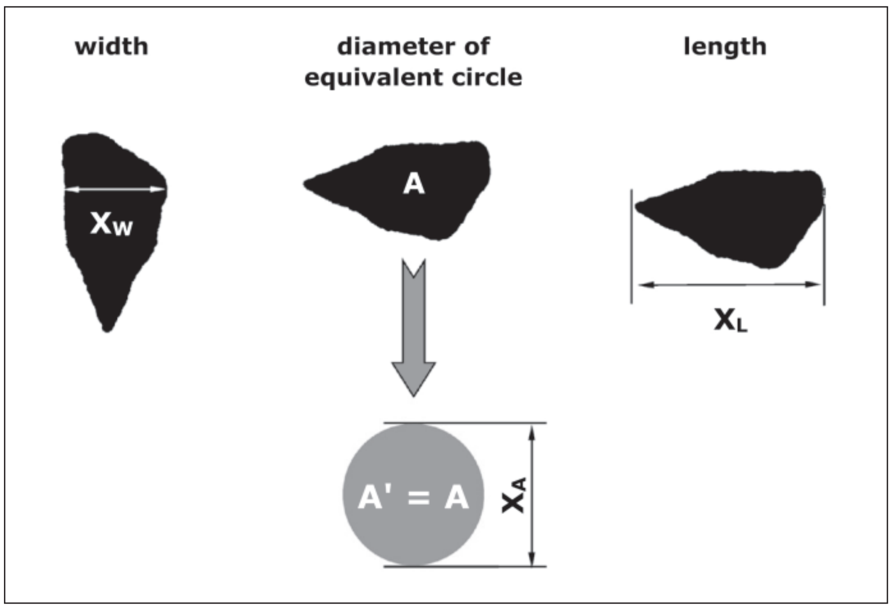

Figure 3. Schematic representation of length (x_L), width (x_W) and diameter (x_A)

Highly Precise Particle Size Measurement Thanks to Shape Analysis

The laser diffraction method is based on the assumption that all particles are spherical. The real particle shape which deviates from the spherical shape changes the light scattering pattern; however, the software cannot transfer these changes to a particular distribution of size and shape. Although it is not possible to differentiate between the length and width of a particle both parameters are included in the calculation of the "particle size". As a result, particle size distribution is often presented wider than it actually is, and with poorer resolution.

If dynamic image processing is used for particle analysis, it is possible to determine the length, width and equivalent diameter separately *(see figure 3)*. Thus it is possible to obtain various size distributions from one measurement, depending on which size definition is considered.

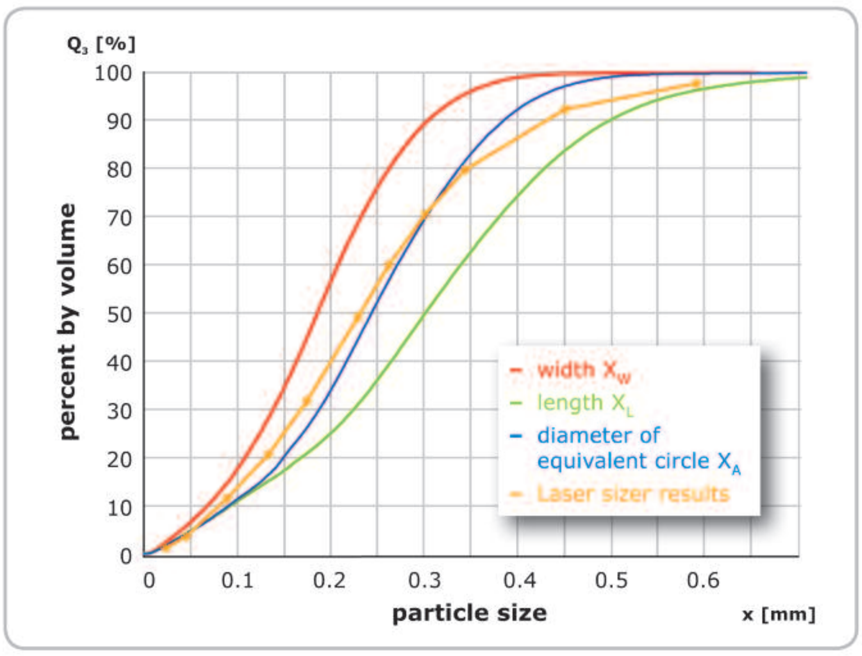

Figure 4. The digital image processing method determines the size distribution with the help of the particle width (x_W, red), the particle length (x_L, green) and the equivalent diameter (x_A, blue). The orange curve represents the results of laser diffraction. The results of image processing are more detailed with a better resolution. The accuracy of the image analysis results is confirmed impressively by sieve analysis and microscopy.

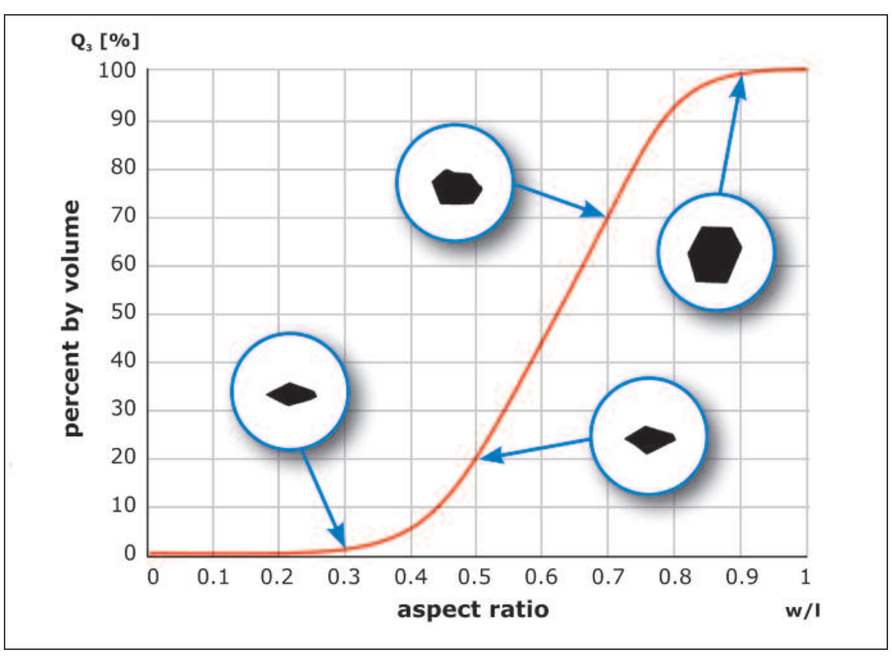

Figure 5. Shape analysis with digital image processing. The graphic shows the width-to-length ratio (w/l) of the sample represented in figure 4. 20% of the particles are twice as long as they are broad, approx. 1 % are three times as long.

Figure 5 clearly shows the deviations between the actual particle shape and the ideal spherical shape the laser scattering method is based on. Spheres have a b/l ratio of 1.0. The majority of particles in the above example have a b/l ratio of < 0.9, for example, they are clearly not spherical. Measuring the particle shape with digital image processing thus leads to a more detailed knowledge of the sample quality.

With dynamic image analysis, just like with laser diffraction, the particles need to pass the field of view individually to ensure that each particle is analysed individually. Agglomerates or particles which stick together give the impression of larger particle sizes. That is why both methods involve dispersion with compressed air or, alternatively, in liquid. The dispersion parameters have to be adjustable in a way that strong agglomerations can be separated without destroying the primary particles. Dynamic image processing provides information about the effectiveness of the dispersion tool as the particle projections are available as pictures at all times.

For particles smaller than 1 micron, laser diffraction remains unrivalled. Image analysis with visible light encounters its physical limits here; as soon as the particle size comes down to the wave length of the light, it is no longer possible to produce sharp pictures of them.

Summary

Dynamic Image Analysis is now available for particle sizes from 1 micron to 3mm, a size range which was previously covered exclusively by laser diffraction. This new method provides the same advantages for fine powders as for larger particles: reliable detection of oversize, high resolution, excellent reproducibility of the particle size results, information about particle shape, as well as easy operation, short measuring times and an intuitive measuring principle. Indirect methods with limited accuracy, such as laser diffraction but also complex optical methods with unreliable statistics, such as microscopy, become increasingly outdated.

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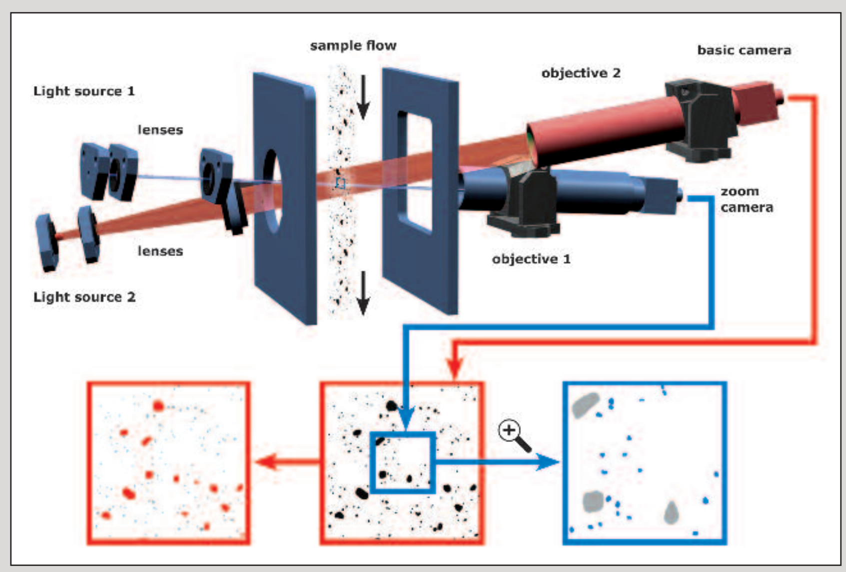
Infobox

Measurement Principle of Dynamic Image Analysis



With Dynamic Image Analysis, the particles move with the help of gravity, compressed air or dispersed in liquid through the measuring field. A light source illuminates them from one side while a camera takes their picture from the other side. The software evaluates the projections of the particles to determine the size distribution of all particles of the sample in a very short time. A few hundred particles per picture are evaluated in real time, more than 275 pictures per second. The maximum dynamic measuring range, i.e. the difference between the smallest and largest detectable particle, is substantially extended by using two aligned cameras. A high resolution camera detects small particles in a small measuring field. A camera with lower resolution but a wider measuring field simultaneously detects the larger particles, allowing for rapid measurement with good statistics.

Anytime, Anywhere Online Analysis

Time is also money in bulk material industries. The response time to manufacturing errors has to be minimised as much as possible. **Haver & Boecker** has developed a Haver CPA system for online measurement of dry and free-falling bulk material in a measuring range from 34µm to 25mm. It is called Haver CPA 2-1 ONLINE. The Haver CPA 2-1 ONLINE is resistant against negative surrounding conditions such as heat, dust and wetness. An integrated cooling cycle system ensures a proven operation even at high temperature operation. An air knife cleans camera and lighting array of dust deposits regularly with an air blast. An absorbent minimises the formation of condensation at high air humidity.

Procedure: The Haver CPA 2-1 ONLINE is ready for continuous measurement. It is connected with a downpipe sampler, which takes samples without any interruption of production. The sampler and CPA device can be activated manually or by a programmable controller with interchangeable memory. The analysis is based on the proven CPA measuring principle: A high-resolution digital line scan camera scans the particles of free-falling bulk materials against the background of an LED-lighting array. Up to 28,000 line scans per second are combined by the CPA software to form an endless data record. The shadow projections are evaluated and displayed in real time (Haver REAL TIME). After measurement the sample is returned to the bulk production quantity. Due to the GigE camera interface a notebook can be placed at a distance of up to 100m from the analysing process.